

# MISSE PEACE Polymers: An International Space Station Environmental Exposure Experiment

Kim K. de Groh and Bruce A. Banks Glenn Research Center, Cleveland, Ohio

Anne M. Hammerstrom, Erica E. Youngstrom, Carolyn Kaminski, Laura M. Marx, and Elizabeth S. Fine Hathaway Brown School, Shaker Heights, Ohio

Jonathan D. Gummow Ohio Aerospace Institute, Brook Park, Ohio

Douglas Wright Cleveland State University, Cleveland, Ohio Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the Lead Center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peerreviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.
- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.
- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.
- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.
- TECHNICAL TRANSLATION. Englishlanguage translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized data bases, organizing and publishing research results . . . even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI Program Home Page at http://www.sti.nasa.gov
- E-mail your question via the Internet to help@sti.nasa.gov
- Fax your question to the NASA Access Help Desk at 301–621–0134
- Telephone the NASA Access Help Desk at 301–621–0390
- Write to:

NASA Access Help Desk NASA Center for AeroSpace Information 7121 Standard Drive Hanover, MD 21076



# MISSE PEACE Polymers: An International Space Station Environmental Exposure Experiment

Kim K. de Groh and Bruce A. Banks Glenn Research Center, Cleveland, Ohio

Anne M. Hammerstrom, Erica E. Youngstrom, Carolyn Kaminski, Laura M. Marx, and Elizabeth S. Fine Hathaway Brown School, Shaker Heights, Ohio

Jonathan D. Gummow Ohio Aerospace Institute, Brook Park, Ohio

Douglas Wright Cleveland State University, Cleveland, Ohio

Prepared for the Conference and Exhibit on International Space Station Utilization-2001 sponsored by the American Institute of Aeronautics and Astronautics Cape Canaveral, Florida, October 15–18, 2001

National Aeronautics and Space Administration

Glenn Research Center

#### Acknowledgments

The authors would like to thank Don Jaworske of GRC for coordinating all the GRC MISSE experiments. We thank Rachel Kamenetzky and colleagues at MSFC for vacuum baking samples. There are many companies who kindly provided samples of polymers, which we sincerely appreciate. We gratefully acknowledge Patty Hunt of Hathaway Brown School for making it possible for the Hathaway Brown students to be a part of this experiment. Finally, we would like to show our sincere appreciation to the MISSE Project Office and Boeing for providing the unique opportunity to be a part of MISSE.

Trade names or manufacturers' names are used in this report for identification only. This usage does not constitute an official endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

Available from

NASA Center for Aerospace Information 7121 Standard Drive Hanover, MD 21076 National Technical Information Service 5285 Port Royal Road Springfield, VA 22100

### MISSE PEACE POLYMERS: AN INTERNATIONAL SPACE STATION ENVIRONMENTAL EXPOSURE EXPERIMENT

Kim K. de Groh and Bruce A. Banks National Aeronautics and Space Administration Glenn Research Center Cleveland, Ohio

Anne M. Hammerstrom, Erica E. Youngstrom, Carolyn Kaminski, Laura M. Marx and Elizabeth S. Fine Hathaway Brown School Shaker Heights, Ohio

> Jonathan D. Gummow Ohio Aerospace Institute Brook Park, Ohio

Douglas Wright Cleveland State University Cleveland, Ohio

#### **Abstract**

Forty-one different polymers are being exposed to the low Earth orbit (LEO) environment on the exterior of the International Space Station (ISS) for one year as part of MISSE (Materials International Space Station Experiment). MISSE is a materials flight experiment sponsored by the Air Force Research Lab/Materials Lab and the National Aeronautics and Space Administration (NASA). A second set of the same polymers is planned to be flown as part of PEACE (Polymer Erosion And Contamination Experiment), a short duration shuttle flight experiment, and therefore these forty-one polymers on ISS are collectively called the MISSE The objective of the MISSE PEACE Polymers. PEACE Polymers experiment is to accurately determine the atomic oxygen (AO) erosion yield of a wide variety of polymeric materials. The polymers range from those commonly used for spacecraft applications, such as Teflon® FEP, to more recently developed polymers, high temperature polyimide such as **PMR** (polymerization of monomer reactants). Additional polymers were included to explore erosion yield dependence upon chemical composition. Details on the specific polymers being flown, flight sample fabrication, and pre-flight characterization techniques will be discussed. The MISSE PEACE Polymers experiment was placed on the exterior of ISS during a spacewalk on August 16, 2001 and is planned to be retrieved in the fall of 2002. The erosion yield data obtained from this experiment will be compared with data from the future short duration experiment PEACE and with predicted results from models developed by a Canadian group that predicts the AO erosion yield of organic materials based on their chemical structure. Having flight data, and comparing flight data with the predictive model results, will be valuable for spacecraft design purposes.

#### Introduction

Polymers such as polyimide Kapton® and Teflon® FEP (fluorinated ethylene propylene) are commonly used spacecraft materials due to their desirable properties such as flexibility, low density, electrical properties, and in the case of FEP, a very low solar absorptance and high thermal emittance. Examples of the use of polymers on the exterior of spacecraft include metallized FEP thermal control materials covering the Hubble Space Telescope, and Kapton solar array blankets and Teflon ePTFE (expanded polytetra-fluorethylene) cable insulation on the International Space Station (ISS).

Polymers on the exterior of spacecraft are exposed to atomic oxygen (AO) in the low Earth orbit (LEO) environment. Atomic oxygen is formed when short wavelength ultraviolet radiation (>5.12 eV, <243 nm) from the Sun photodissociates molecular oxygen in the upper atmosphere.<sup>1</sup> Although AO is the predominant

species in LEO (below ≈1,000 km)<sup>2</sup>, these neutral oxygen atoms have mean free paths on the order of 10<sup>4</sup> m at 400 km, resulting in extremely low probabilities of re-association. As a spacecraft orbits the Earth it travels with a velocity on the order of 7.7 km/sec and it rams into the atmospheric oxygen (hence the term ram AO). The flux of AO at International Space Station (ISS) altitudes is approximately 1.0 x 10<sup>14</sup> atoms/cm<sup>2</sup> sec for normal incident ram surfaces, and the average energy of an oxygen atom impacting spacecraft at ram velocities is 4.5 eV.<sup>3</sup> A number of processes can take place when an oxygen atom strikes a spacecraft surface at orbital velocities. These include chemical reaction with surface atoms or adsorbed molecules, elastic scattering, scattering with partial or full thermal accommodation, recombination, or excitation of ram species.<sup>4</sup> Because the oxidation product for most polymers is a gas, AO erosion results.

Atomic oxygen erosion of polymers in LEO is a serious threat to spacecraft durability. For example, more than 0.0127 cm (0.005") thickness of Kapton and Mylar sheets were eroded away after 5.8 years in LEO on the leading edge, or ram AO surface, of the Long Duration Exposure Facility (LDEF).<sup>5</sup> AO fluence for the leading edge of LDEF was 8.99 x 10<sup>21</sup> atoms/cm<sup>2.6</sup> In addition to the erosion of the Kapton and Mylar films, ≈1 ply (0.0127 cm or 0.005") of graphite-epoxy and 0.0025 cm (0.001") of Teflon FEP were also eroded away after 5.8 years on LDEF's leading edge.<sup>5</sup> Figure 1 shows a fluoropolymer (polychlorotrifluoroethylene) that was exposed to near normal incidence ram AO on the leading edge of LDEF. A cone-like or carpet type morphology developed, which is characteristic of directed AO erosion for materials with gaseous oxidation products. Protective coatings can be effective in preventing AO erosion, yet oxidation erosion of the underlying polymer can still occur at pinhole and scratch defects through AO undercutting erosion.<sup>7-9</sup>

In addition to the obvious potential degradation to the structural ability of the polymers such as the support of photovoltaic cells on the ISS solar array, AO is a threat to other materials properties. For example, the thermal emittance of thin polymer thermal control materials is dependent upon the thickness of the polymer. Thus, erosion of the polymer by AO can result in a reduced thermal emittance capability, which would give rise to increases in spacecraft temperature. It is therefore essential to understand the AO erosion yield (E, the volume loss per incident oxygen atom) of polymers being considered in spacecraft design. Procedures have been established for ground laboratory

AO interaction evaluation of materials for space applications. Although ground laboratory procedures have been established and are used for erosion yield determination, actual in-space data is more reliable and therefore more desirable than results of ground tests.

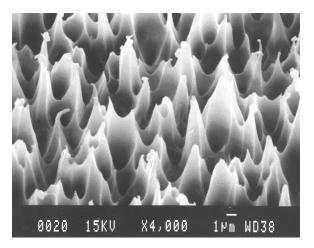


Figure 1. This fluoropolymer was located on the leading edge of LDEF and shows the recession morphology that is typical of directed ram AO erosion.

National Aeronautics and Space Administration (NASA) Glenn Research Center (GRC) has been given the opportunity to expose forty-one different polymers to the LEO environment on the exterior of the ISS for one year as part of the MISSE (Materials International Space Station Experiment) project. MISSE is a passive materials flight experiment sponsored by the Air Force Research Lab/Materials Lab and NASA. A second set of the same polymers will be flown at a later date as part of PEACE (Polymer Erosion And Contamination Experiment), a short duration shuttle flight experiment, and therefore these forty-one polymers on ISS are collectively called the MISSE PEACE Polymers. The purpose of the MISSE PEACE Polymers experiment is to accurately determine the AO erosion yield of a wide variety of polymeric materials exposed for an extented period of time to the LEO space environment. This paper reviews the specific polymers being flown in the MISSE PEACE Polymers experiment, the flight sample fabrication procedures, and pre-flight characterization techniques that were used. The erosion yield data obtained from the MISSE PEACE Polymers experiment will be compared with data obtained from the short duration experiment PEACE and with ground data. The LEO erosion yield data will be compared with results from an analytical model developed by the Integrity Testing Laboratory Inc. that predicts the AO erosion yield of organic materials based on their chemistry. 11 Having

the erosion yield data for many different polymers that are all characterized and exposed to space under identical conditions, and having space data to compare with the predictive model, will be valuable for spacecraft design purposes in the future.

#### **Erosion Yield Measurements**

#### **Erosion Yield Determination**

The most common technique for determining the erosion yield of flight samples is through mass loss measurements. These measurements are made by obtaining mass measurements of the sample before and after flight. The erosion yield of the sample (E<sub>S</sub>) is calculated through the following equation:

$$E_S = \frac{\Delta M_S}{(A_S \rho_S F)} \tag{1}$$

where

 $E_S$  = erosion yield of flight sample (cm<sup>3</sup>/atom)

 $\Delta M_s = mass \ loss \ of the flight sample (g)$ 

 $A_s$  = surface area of the flight sample exposed to atomic oxygen attack (cm<sup>2</sup>)

 $\rho_s = density \ of \ sample \ (g/cm^3)$ 

F = fluence of atomic oxygen (atoms/cm<sup>2</sup>)

The AO fluence (F) can be determined through the mass loss of a Kapton witness sample because Kapton has a well characterized erosion yield in the LEO environment. Therefore, the AO fluence can be calculated using the following equation:

$$F = \frac{\Delta M_K}{\left(A_K \rho_K E_K\right)} \tag{2}$$

where

 $\Delta M_K = mass \ loss \ of \ Kapton \ witness \ sample \ (g)$ 

 $A_K$  = surface area of Kapton witness sample exposed to atomic oxygen (cm<sup>2</sup>)

 $\rho_K = density of Kapton witness sample (1.42 g/cm^3)$   $E_K = erosion yield of Kapton witness sample$  $(3.0 \times 10^{-24} \text{ cm}^3/\text{atom})$ 

Thus

$$E_S = E_K \frac{\Delta M_S A_K \rho_K}{\Delta M_K A_S \rho_S} \tag{3}$$

#### Rehydration/Dehydration Issues

One of the critical issues with obtaining accurate erosion yield data from mass measurements is making sure that dehydrated mass measurements are taken. Many polymer materials, such as Kapton, are very hygroscopic (absorbing up to 2% of their weight in moisture) and can fluctuate in mass significantly with humidity and temperature. Therefore, for accurate mass loss measurements to be obtained, it is necessary that the samples be fully dehydrated (i.e. in a vacuum desiccator) prior to measuring the mass for both preflight and post-flight.

#### Polymeric Materials

As mentioned previously, the specific polymers chosen for the MISSE PEACE Polymers experiment are essentially the same polymers that will be flown as part of the shuttle flight experiment PEACE. These polymers represent a wide range of polymeric materials and bonding types. Included are those commonly used for spacecraft applications, such as Teflon FEP, to more recently developed polymers, such as high temperature polvimide PMR (polymerization of monomer reactants). Polymers such as polyethylene oxide and cellulose acetate are also included based solely on their chemical composition to provide LEO erosion yield data for modeling purposes. Table 1 provides the list of the 41 MISSE PEACE Polymers materials and their associated polymer abbreviation and common trade names. Also provided in the table is the MISSE serial number for each sample. Two polyimide Kapton H samples have been included to serve as fluence calibration witness samples.

#### Sample Fabrication & Characterization

#### Flight Sample Dimensions & Fabrication

The MISSE PEACE Polymers samples are approximately 1" (2.54 cm) diameter disks (0.995 ± 0.005 in, or  $2.527 \pm 0.013$  cm). The flight hardware has sample hole areas that are  $1.020 \pm 0.005$ " (2.591  $\pm$ 0.013 cm) diameter with an exposure opening, and therefore an exposure diameter, of  $0.813 \pm 0.010$ "  $(2.065 \pm 0.025 \text{ cm})$ , see Figure 2. The polymers were most often obtained in thin film form, typically ranging from 1 to 20 mils thick (0.0025 cm to 0.051 cm) and were punched into the ≈1" diameter disks using a double bow punch cutter and an Arbor press.

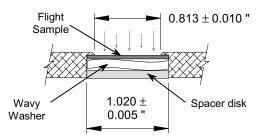


Figure 2. Cross-section diagram of the MISSE PEACE Polymers sample holder (without the backing plate).

Table 1. MISSE PEACE Polymers List.

MISSE	Material	Abbrev.	Trade Names
Serial #			
-2 -E5 -6	Acrylonitrile butadiene styrene	ABS	Cycolac
-2 -E5 -7	Cellulose acetate	CA	Clarifoil; Tenite Acetate; Dexel
-2 -E5 -8	Poly-(p-phenylene terephthalamide)	PPD-T	Kevlar 29 fabric
-2 -E5 -9	Polyethylene	PE	_ ,, ,
-2 -E5 -10	Polyvinyl fluoride	PVF	Tedlar TTR10SG3
-2 -E5 -11	Crystalline polyvinylfluoride w/white pigment	PVF	White Tedlar TW10B53
-2 -E5 -12	Polyoxymethylene; acetal; polyformaldehyde	POM	Delrin (Natural)
-2 -E5 -13	Polyacrylonitrile	PAN	Barex 210
-2 -E5 -14	Diallyl diglycol and triallyl cyanurate	ADC	CR-39, Homalite H-911
-2 -E5 -15	Polystyrene	PS	Trycite 1000/Trycite Dew
-2 -E5 -16	Polymethyl methacrylate	<b>PMMA</b>	Plexiglas; Lucite; Acrylite (Impact Mod.)
-2 -E5 -17	Polyethylene oxide	PEO	Alkox E-30
-2 -E5 -18	Poly(p-phenylene-2 6-benzobisoxazole)	PBO	Balanced Biaxially Film
-2 -E5 -19	Epoxide or epoxy	EP	Hysol EA 956
-2 -E5 -20	Polypropylene	PP	Type C28
-2 -E5 -21	Polybutylene terephthalate	PBT	GE Valox 357
-2 -E5 -22	Polysulphone	PSU	Thermolux P1700-NT11; Udel P-1700
-2 -E5 -23	Polyeurethane	PU	Dureflex PS 8010
-2 -E5 -24	Polyphenylene isophthalate	PPPA	Nomex Aramid Paper Type 410
-2 -E5 -25	Graphite	PG	Pyrolytic Graphite
-2 -E5 -26	Polyetherimide	PEI	Ultem 1000
-2 -E5 -27	Polyamide 6 or nylon 6	PA 6	Akulon K; Ultramid B
-2 -E5 -28	Polyamide 66 or nylon 66	PA 66	Maranyl A; Zytel
-2 -E5 -29	Polyimide	PI	LaRC CP1 (CP1-300)
-2 -E5 -30	Polyimide (PMDA)	PI	Kapton H
-2 -E5 -31	Polyimide (PMDA)	PI	Kapton HN
-2 -E5 -32	Polyimide (BPDA)	PI	Upilex-S
-2 -E5 -33	Polyimide (PMDA)	PI	Kapton H
-2 -E5 -34	High temperature polyimide resin	PI	PMR-15
-2 -E5 -35	Polybenzimidazole	PBI	Celazole PBI
-2 -E5 -36	Polycarbonate	PC	PEEREX 61 (P61)
-2 -E5 -37	Polyetheretherkeytone	PEEK	Victrex PEEK 450
-2 -E5 -38	Polyethylene terephthalate	PET	Mylar A/200
-2 -E5 -39	Chlorotrifluoroethylene	CTFE	Neoflon CTFE M-300; Kel-F
-2 -E5 -40	Halar ethylene-chlorotrifluoroethylene	ECTFE	Halar
-2 -E5 -41	Tetrafluorethylene-ethylene copolymer	ETFE	Tefzel ZM
-2 -E5 -42	Fluorinated ethylene propylene	FEP	Teflon FEP (round robin)
-2 -E5 -43	Polytetrafluoroethylene	PTFE	Chemfilm DF 100
-2 -E5 -44	Perfluoroalkoxy copolymer resin	PFA	Teflon PFA CLP (200 CLP)
-2 -E5 -45	Amorphous Fluoropolymer	AF	Teflon AF 1601
-2 -E5 -46	Polyvinylidene fluoride	PVDF	Kynar 740
-2 -E3 -40	i organizmache muonae	1 1 1 1 1 1	Kynai /40

Several samples did not come in film form. Diallyl diglycol and triallyl cyanurate (ADC, sample -2-E%-14), typically used as lens material, had to be ordered from a manufacturer in ≈1" diameter, 31 mil thick (0.079 cm) samples. Pyrolytic graphite (PG, sample -2-E5-25) was also ordered in ≈1" diameter pieces, 80 mil thick (0.203 cm). Hysol EA 956 epoxy (EP, sample -2-E5-19) was purchased as a two-part kit. Flight samples were fabricated by mixing and curing the epoxy, and then carefully sawing out 1" diameter samples from brittle sheets of cured epoxy. The thickness of the flight sample is ≈91 mil thick (0.231 cm). Polyethylene oxide (PEO, -2-E5-17) was purchased as a powder and fabricated into sheet material by pressing the powder with heated plates using a Carver Laboratory Press. To keep the PEO from sticking to the plates, two sheets of Kapton were placed in between the press plates and the PEO powder. The PEO was pressed at 23,000 lbs (10.5 metric tons) for 5-10 seconds while simultaneously applying heat. The exact temperature of heating is not known, but the press heats to 500 °F and approximately 40% of the power was used. After pressing, when completely cooled, the Kapton sheets were carefully separated from the PEO. This resulted in a sheet of PEO between 29 and 37 mils thick, from which 1" diameter samples were punched out. High temperature polyimide resin, PMR-15 (PI, sample -2-E5-34), was fabricated by the Polymers Branch at GRC in 1" diameter, 12 mil (0.030 cm) thick pieces. Poly-(p-phenylene terephthalamide), also known as Kevlar 29 (PPD-T, -2-E5-8), was obtained in fabric form. Flight samples were cut out and the fabric samples were carefully wrapped in Al disks to protect the edges from fraying or loosing small filaments. The extreme edge on the exposed side was covered with Al so that only the Kevlar would be exposed to AO in the flight hardware (knowing the exposed area is important in calculating the erosion yield using mass loss measurements). Polyphenylene isophtalate (PPPA), also known as Nomex<sup>®</sup>, was supplied as 2 mil (0.005 cm) thick sheets of paper, from which flight samples were easily punched out. All other polymers were supplied in thin film form.

#### Sample Stacking

The expected AO fluence for a 1-year exposure on ISS is 3.28 x 10<sup>21</sup> atoms/cm<sup>2</sup> for directed AO exposed surfaces, based on a mission launch date of June 2001, a 400 km circular orbit and a 51.6 degree inclination. Many of the thin film polymers will be completely eroded away after 1 year of exposure and the experiment may not be retrieved on time.

Therefore, depending on the anticipated erosion yield of the polymer and the polymer thickness, stacking of several sample layers was necessary. Stacking of the samples is complicated by the fact that increasing the mass of the sample causes a decrease in the sensitivity of the mass change that occurs. Therefore, one would ideally want to measure the lightest sample possible, before and after flight, so the mass loss is a significant percent of the total mass of the sample. Keeping this in mind, two different values were used in determining the number of sample layers needed for flight. The first value was how many sample layers should be weighed, and the second was how many total layers should be flown.

It was decided that enough sample material should be stacked and weighed to survive an AO exposure period of 1.5 years. Originally, the MISSE experiment was planned as a 1-year mission, and so it was decided to choose a mission time slightly longer than expected for sample weighing (hence 1.5 years for an expected 1-year mission). Therefore, based on the expected AO fluence for a 1.5 year mission which was computed to be 4.55 x 10<sup>21</sup> atoms/cm<sup>2</sup>, and the anticipated erosion yield of the polymer, the number of layers for each sample that needed to be weighed was determined. The total number of layers to be stacked was decided based on surviving a 3-year mission, because flight missions are not always retrieved on the original planned date. For example, the LDEF was planned as a 1-year mission, but instead was exposed to the LEO environment for 5.8 years.<sup>12</sup> The AO fluence for a 3-year mission was 9.1 x 10<sup>21</sup> atoms/cm<sup>2</sup> (in this case, simply based on twice the 1.5-year fluence); therefore the total number of samples to be stacked as "one flight sample" was calculated based on surviving this fluence. The additional "stacked" samples were always placed behind the "weighed" sample stack in the flight hardware. When the experiment is retrieved, only the remaining layers of the original stack that was weighed will be re-weighed to determine the mass loss of the sample. The 3-year fluence estimate used for calculating the sample stack was higher than what is actually expected because of decreases in the solar activity expected based on the time within the 11 year solar cycle.

For example, the erosion yield for Teflon FEP is  $3.37 \times 10^{-25} \text{ cm}^3/\text{atoms}$ , <sup>13</sup> so the predicted thickness loss after 1.5 years will be 0.6 mils based on the simple equation:  $E(cm^3/atom) \times fluence(atoms/cm^2) = thickness loss (cm)$ . The thickness of one flight sample of Teflon FEP is 2.0 mils, so only one sample needed to be weighed before flight and no layers needed to be

stacked behind it (only 1.2 mils expected to be eroded after 3 years of space exposure). Another example is cellulose acetate (CA), which has a predicted erosion yield of 6.8 x 10<sup>-24</sup> cm<sup>3</sup>/atom.<sup>14</sup> The estimated thickness loss for 1.5 years in LEO is 12.2 mils (0.031 cm) and the sample thickness is only 2 mil (0.005 cm) therefore 7 sample layers need to be stacked for mass measurements. A total of 24.4 mils (0.062 cm) is expected to be eroded away after 3 years, so 13 total layers needed to be stacked for flight (the 7 weighed samples + 6 additional samples stacked behind). In all, a total of 205 individual samples layers were flown as part of the 41 MISSE PEACE Polymers.

For each flight sample, an identical back-up sample (or sample stack) was prepared and characterized. It is important to have back-up samples available so that if problems occur, such as while loading samples into the flight hardware in a clean room environment, a second flight-ready sample is available to be used when needed. All individual sample layers were carefully marked at the edges of the samples indicating sample ID, orientation (front), vacuum baked or not, and flight or back-up sample.

#### Outgassing & Vacuum Heat Treatment

Samples to be flown in the space environment need to meet outgas requirements as outlined in ASTM E 595, the Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment.<sup>15</sup> This test method evaluates, under carefully controlled conditions, the changes in the mass of a test specimen on exposure under vacuum to a temperature of 125 °C (total mass loss (TML)), and the mass of those products that leave the specimen and condense on a collector plate at a temperature of 25 °C (collected volatile condensable materials (CVCM)).<sup>15</sup> The criteria used in acceptance and rejection of materials under ASTM E-595 is to be determined by the user, based upon specific component and system requirements. 15 Historically, the screening levels are <1.00% TML and <0.10% CVCM.<sup>16</sup>

MISSE management decided that material being flown as part of MISSE should meet the historical screening levels of <1.00% TML and <0.10% CVCM. Many spacecraft materials have been tested for TML and CVCM using ASTM E 595 methods and the data are available in the literature. Ten of the 41 PEACE polymers did not meet the outgas requirements based on previous testing or because they had not been tested, and it was decided that these polymers could be flown

if they were vacuum baked prior to flight to remove volatile products. Vacuum baking was conducted at the NASA Marshall Space Flight Center (MSFC) at a pressure of 5 x 10<sup>-7</sup> torr (7 x 10<sup>-5</sup> Pa). Ideally samples were to be vacuum baked for 24 hours at 125°C (similar to ASTM E 595), but some polymers were heated at lower temperatures due to low maximum operating temperatures. Table 2 lists the vacuum-baked samples, their maximum operating temperature and the vacuum heat-treating temperature and time.

The polymer PBI (sample -2-E5-35) curled severely at its edges during the vacuum heat treatment. For this polymer, 4 individual sample layers needed to be stacked to survive a 3-year exposure. Therefore the 4 layers were stacked together (after mass measurements were obtained) and held flat by mounting them in an Al holder, similar to the one used for the Kelvar fabric. This was necessary to make sample handling for loading in the flight hardware easy.

#### Pre-flight Dehydrated Mass Measurements

All pre-flight mass measurements were obtained for dehydrated sample stacks. For the samples that vacuum baked, the dehydrated measurements were obtained at GRC shortly after vacuum baking at MSFC. All sample stacks were dehydrated in a vacuum desiccator maintained at a pressure of 60-100 mtorr with a mechanical roughing pump. Typically 5 flight samples and their corresponding back-up samples were weighed in a group (a total of 10 stacks of samples). The flight and back-up samples were placed in the vacuum desiccator in a particular order and left under vacuum for a minimum of 4 days. The exact time that samples were under vacuum was recorded. Typically, the flight samples, followed by the back-up samples, were In order to maintain each sample under vacuum while weighing other samples, the vacuum desiccator would be put back under vacuum immediately after an individual sample was removed. The time the sample was removed from the desiccator was carefully recorded along with the times it was weighed (a total of 3 mass readings were obtained and averaged). Previous tests conducted at GRC showed that the mass of a dehydrated sample was not adversely affected, if the desiccator was opened and quickly closed again prior to that sample being weighed. This allows multiple samples to be dehydrated and weighed at a time. The samples were weighed using either a Mettler Balance or a Sartorius Balance depending on their total weight. Records of the following were kept: the sequence of sample weighing, the number of samples in the stack being weighed, the time under vacuum prior to weighing, the temperature and humidity in the room, the time a sample was taken out of the desiccator and the time of each weighing. An example of data recorded in a Sample Weight Table is provided in Table 3. Only the flight samples are listed in this particular table, although the data for the back-up samples are included in the original table. The exact sequence and procedure will be repeated with the same samples after retrieval from the space environment.

Table 2. List of Vacuum Heat Treated MISSE PEACE Polymers.

MISSE Serial #	Material	Abbreviation/ Trade Name	Max. Operating Temperature (C)	Vacuum Heat Treatment
-2 -E5 -6	Acrylonitrile butadiene styrene	ABS/Cycolac	105	24 hrs @ 90C
-2 -E5 -7	Cellulose acetate	CA/Clarifoil	230-245	68.75 hrs @ 128C
-2 -E5 -8	Poly-(p-phenylene terephthalamide) fabric in Al foil	PPD-T/Kevlar fabric	149-177	24 hrs @ 128C
-2 -E5 -10	Polyvinyl fluoride	PVF/Tedlar	107	36 hrs @ 100C
-2 -E5 - 13	Polyacrylonitrile	PAN/Barex	200-210	24.25 hrs @ 126C
-2 -E5 - 17	Polyethylene oxide	PEO/Alkox	65	24 hrs @ 60C
-2 -E5 -24	Polyphenylene isophthalate	PPPA/Nomex paper	220	24 hrs @ 125C
-2 -E5 -27	Polyamide 6 or nylon 6	PA 6/Akulon	98	24 hrs @ 90C
-2 -E5 -28	Polyamide 66 or nylon 66	PA 66/Maranyl	120	24 hrs @ 90C
-2 -E5 -35	Polybenzimidazole	PBI/Celazole	343	24 hrs @ 125C

Table 3. Sample Weight Table.

MISSE Sample #	Material	# of Layers	Sample Weigh Order	Time out of Desiccator	Time	Mass (mg)	Avg. Weight (mg)
2 55 40					10:13:30	126.11	
-2-E5-40 F	Halar	2	1	10:10:00 AM	10:14:30	126.125	126.118
1					10:15:20	126.118	
2 E5 22					10:20:00	116.021	
-2-E5-32 F	Upilex	6	2	10:18:10 AM	10:21:10	116.006	116.016
1					10:22:00	116.022	
0 F5 21					10:30:15	183.432	
-2-E5-31 F	Kapton HN	2	3	10:27:30 AM	10:31:15	183.482	183.483
Г					10:32:00	183.535	
2.55.20					10:37:20	187.964	
-2-E5-30 F	Kapton H	2	4	10:36:45 AM	10:38:20	188.048	188.059
Γ					10:39:20	188.164	
2.55.20					10:46:45	123.707	
-2-E5-28 F	PA 66	4	5	10:45:00 AM	10:47:45	123.75	123.716
Г					10:48:30	123.691	

#### Flight Sample Mounting (Tray E5)

The MISSE PEACE Polymers were designated to reside in sample tray E5, which holds a total of 46 - 1" diameter flight samples. These samples start in the top row, 6<sup>th</sup> sample position from the left (i.e. ABS has serial number -2-E5-6, which represents a sample in MISSE Passive Experiment Container (PEC) 2, Tray E5, sample position 6). Five other GRC samples (4 DC

93-500 silicone samples and 1 AO scattering chamber) are also on this tray in positions -2-E5-1 through -2-E5-5. Sample positions for the polymers were chosen based on their anticipated erosion yields or by grouping samples by polymer family. For example, expected high erosion yield samples were located next to the silicone samples because these samples are less likely to be effected if cross-contamination occurs from the

silicones. The polyimides were placed together, as were the fluoropolymers. The 46 samples were loaded into tray E5 in a clean room environment at GRC with help from student investigators. Samples were loaded upside down into the back of the flight hardware. Properly cleaned flight-quality wavy washers and disk spacers were added behind the sample stacks to fill in the extra space as needed. A backing plate was placed

on the back after all samples and spacers were in place. Black lights were used for examining the loaded samples for dust identification and removal purposes. Dust was removed using a slow nitrogen gas flow, or tweezers if necessary. Figure 3 shows the 41 MISSE PEACE Polymers samples in tray E5 along with the 5 other GRC samples, ready for assembly into the MISSE flight hardware.



Figure 3. GRC's 41 MISSE PEACE Polymers loaded into sample tray E5. Samples with circles have expected high erosion yields, samples within the solid line block are from the polyimide family and samples within the dashed line block are from the fluoropolymer family.

### <u>Materials International Space Station Experiment</u> (MISSE)

Managed by Langley Research Center (LaRC) in Hampton, Va., MISSE is a collaborative effort among NASA centers, the U.S. Air Force and private industry. It is a cooperative experiment involving Principle Investigators from Boeing Phantom Works, the Materials Laboratory at the Air Force Research Laboratory, NASA's LaRC, MSFC and GRC. MISSE will test the space environmental durability of hundreds of samples ranging from lubricants to solar cell technologies. MISSE consists of 1-year and 3-year passive exposure trays to be exposed to AO and solar radiation (in addition to other environmental exposures), or solar radiation with no AO exposure, on the exterior of ISS.

The MISSE PEACE Polymers are located on PEC 2-Tray 1, one of the 1-year AO and solar exposure trays. Figure 4 shows PEC 2 with both the AO and solar exposure tray (Tray 1, on the right) and the solar exposure tray (Tray 2, on the left). These trays fold together with the samples protected inside the PEC for transportation and launch, and fold back-to-back with the samples facing out in the space environment. The 1-year MISSE trays (PEC 1 & 2) have been transported

to the ISS, and clamped to exterior Quest Airlock handrails on August 16, 2001 during a space walk as part of the STS-105 shuttle mission. Figure 5 is a photograph of MISSE PEC 2 on the ISS Quest Airlock just after attachment. The MISSE PEACE polymers tray is visible in this photograph. Figure 6 is a photograph of ISS that shows the locations of both MISSE PEC 1 and PEC 2. Retrieval of the MISSE 1-year trays is planned for the fall of 2002.

#### Comparison to the Shuttle Experiment PEACE

PEACE is a short-duration passive experiment, currently planned to fly as a shuttle flight experiment to be exposed to the LEO environment from the shuttle bay. PEACE is a collaborative effort between GRC and Hathaway Brown School, a private all girls high school. The technical objectives of PEACE are 1) to measure the LEO AO erosion yields of a wide variety of polymer materials, and 2) to validate a method for identifying sources of silicone contamination which occur in the Shuttle bay and on spacecraft in LEO. 19 The AO erosion yields of forty-two (42) different polymers (40 of which are the same as those in the MISSE PEACE Polymers experiment) will be determined using two independent techniques based on mass loss and recession depth. One-inch diameter

samples will be used for obtaining mass loss. One-half inch diameter samples will be protected with isolated AO durable particles (such as salt crystals or mica dust) during exposure to the space environment. After flight, the protective particles will be removed and atomic force microscopy (AFM) will be used to measure the erosion or recession depth from protected mesas.<sup>20</sup> Kapton witness samples in both one inch and one-half inch diameter sizes will be installed at various locations among the polymer samples for use in determining AO fluence. For low fluence exposure experiments obtaining the erosion yield by the recession depth technique, using thin protective particles (of NaCl or mica) is more accurate and thus has the advantage of providing meaningful erosion yield data for short duration experiments. For example, for a fluence of 2 x 10<sup>19</sup> atoms/cm<sup>2</sup> the probable error of the erosion yield is 6.72% for the mass loss technique and 4.41% for the AFM recession technique for a 10 µm thick protective particle.20

The polymers to be flown as part of PEACE will be from the same batch of material being flown on MISSE. PEACE is planned as a short duration experiment to be characterized with a technique to provide meaningful low fluence erosion yield data; therefore, through PEACE and MISSE, both low and high fluence erosion yield data will be available for these 40 polymers. It will be very beneficial for spacecraft design and durability purposes to be able to

compare the erosion yields for high fluence exposures with those measured for short fluence exposures. There has been some evidence that AO erosion may be dependent on duration for some polymers. This is because ultraviolet radiation and energetic particle interaction may introduce a synergistic effect with AO erosion for long term flights, possibly due to reaching a threshold in radiation damage for the longer duration exposures.

#### Comparison to Predictive Model Data

Erosion yield prediction methods have been developed within the US and Canada and predictions have been generated for many of the same 40 polymers based on their chemical composition and structure. 11,14 The MISSE PEACE polymers erosion yield data will be directly compared with predictions made by Integrity Testing Laboratory, Inc. as contracted by GRC. 14 These predictions are made based on predictive models, developed for interaction of polymers with the LEO environment, and using information about the chemical composition, structure, and densities, as well as experimental data for Oxygen Index. 11 Obtaining actual LEO erosion yield data on the same polymers would provide the necessary data that would either validate the predictive methods or lead to the necessary improvements to be able to predict in-space durability of new materials without requiring in-space testing.



Figure 4. MISSE PEC 2 with Tray 1 on the right and Tray 2 on the left. 18



Figure 5. During a spacewalk on August 16, 2001, astronaut Patrick Forrester installs MISSE PEC 2 on the ISS Quest Airlock [NASA photo STS105E5302].

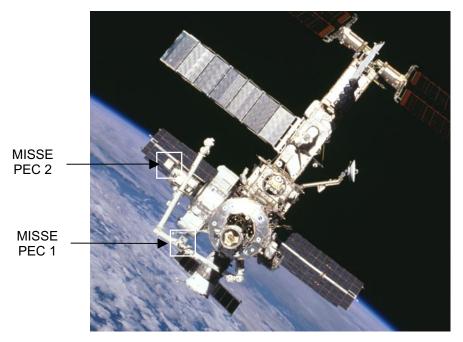


Figure 6. Photograph of ISS taken on August 20, 2001, showing the locations of MISSE PEC 1 and PEC 2 [NASA photo STS105-713-037].

#### Summary & Conclusions

Forty-one different polymers are being exposed to the LEO environment for approximately one year as part of MISSE, the first exterior experiment on the ISS hull. A second set of the same polymers will be flown as part of PEACE, a short duration shuttle flight experiment, and therefore these forty-one polymers on ISS are collectively called the MISSE PEACE Polymers. The purpose of the MISSE PEACE

Polymers experiment is to accurately determine the AO erosion yield of a wide variety of polymeric materials, all characterized and exposed under identical conditions. The polymers include those commonly used in spacecraft applications to polymers chosen strictly based on their chemistry for modeling purposes.

The MISSE PEACE Polymers samples were fabricated into 1" (2.54 cm) diameter disks. The thickness of the polymers typically ranges from 1 to 10

mils thick (0.0025 cm to 0.0254 cm). Estimated erosion yields were used to determine the necessary number of layers of material that would be required to survive a 1 1/2-year mission for mass measurements. Carefully documented dehydrated pre-flight mass measurements were obtained for flight and back-up samples. Because the date of retrieval of MISSE is not guaranteed, additional layers of samples were stacked behind the weighed flight samples to ensure that there would be material left after a 3-year exposure. This resulted in flight samples consisting of between 1 to 13 individual film layers. Numerous polymers needed to be vacuum baked prior to flight because they did not meet outgas requirements. Details of the specific polymers being flown, flight sample fabrication and pre-flight techniques used were discussed.

The erosion yield data obtained from this experiment will be compared with data from the short duration experiment PEACE. The LEO erosion yield data will then be compared to erosion yield values obtained from a predictive model developed by a Canadian group that predicts the AO erosion yield of organic materials based on their chemical structure. Having the erosion yield data for many different polymers, all characterized and exposed to space for a long duration under identical conditions, and having space data to compare with the predictive model results, will be valuable for future spacecraft design purposes.

#### References

- 1. Banks, B. A., Mirtich, M. J., Sovey, J. S., Nahra, H., and Rutledge, S. K., NASA CP-3109, Vol. 2, 1990, pp. 179-184.
- 2. *U.S. Standard Atmosphere*, 1976, U.S. Govt. Printing Office, Washington, D.C., 1976, p. 30.
- 3. Banks, B. A., Rutledge, S. K., Auer, B. M., and DiFilippo, F., *Materials Degradation in Low Earth Orbit (LEO)*, TMS Society, 1990, pp. 15-33.
- 4. Gregory, J. C., Proceedings of the NASA Workshop on Atomic Oxygen Effects, Nov. 10-11, 1986, JPL 87-14, 1987, pp. 29-30.
- 5. Stein, B. A. and Pippin, H. G., NASA CP 3134, Part. 2, pp. 617-641.
- 6. Silverman, E. M., NASA CR 4661, Part 1, August 1995.

- 7. de Groh, K. K. and Banks, B. B., J. Spacecraft and Rockets, Vol. 31, No. 4, 1994, pp. 656-664.
- 8. de Groh, K. K., Dever, J. A., McCollum, T. A., Rodriguez, E., Burke, C. and Terlep J. A., Solar Engineering 1992, Vol.2, ASME, pp. 775-782.
- 9. Banks, B. A., de Groh, K. K., Auer, B. M. and Gebauer, L., NASA CP 3257, 1994, pp.143-158.
- 10. ASTM E 2089-00 Standard Practices for Ground Laboratory Atomic Oxygen Interaction Evaluation of Materials for Space Applications, *Annual Book of ASTM Standards*, 2000.
- 11. Kleiman, J., Iskanderova, Z., Banks, B. A., de Groh, K. K., and Sechkar, E. A., Proceedings of the 8<sup>th</sup> International Symposium on Materials in a Space Environment, Arcachon, France, June 5-9, 2000.
- 12. Levine, A. S., NASA CP 3134, Part. 2, 1991, pp. iii-iv.
- 13. Banks, B. A., "The Use of Fluoropolymers in Space Applications," Ch. 4 in *Modern Fluoropolymers: High Performance Polymers for Diverse Applications*, John Wiley & Sons, Ltd., 1997.
- 14. "Prediction of Erosion of Polymer-Based Materials by Atomic Oxygen in LEO," Integrity testing Laboratory Inc. Final Report, GRC Contract #C-72917-G, 1998.
- 15. ASTM E 595-84 Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment, *Annual Book of ASTM Standards*, 1984.
- 16. Walter, N. A,. & Scialdone J. J., NASA RP 1124-Rev. 4, June 1997.
- 17. MISSE Fact Sheet, <a href="http://www.msfc.nasa.gov/NEWSROOM/background/facts/misse.html">http://www.msfc.nasa.gov/NEWSROOM/background/facts/misse.html</a>, 2001.
- 18. MISSE home page, <a href="http://misse1.larc.nasa.-gov/index.html">http://misse1.larc.nasa.-gov/index.html</a>, 2001.
- 19. Banks, B. A., de Groh K. K., Baney-Barton, E., Sechkar, E. A., Hunt, P., Willoughby, A., Bemer, M., Hope, S., Koo, J., Kaminski, C. & Youngstrom, E., NASA TM-1999-209180, May 1999.
- 20. de Groh, K. K., Banks, B. A., Clark, G. W., Hammerstrom A., Youngstrom, E., Kaminski, C., Fine, E. & Marx, L. "A Sensitive Technique Using Atomic Force Microscopy to Measure the Low Earth Orbit Atomic Oxygen Erosion of Polymers." NASA TM-2001-211346.

#### REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

AGENCY USE ONLY (Leave blank)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED			
Transition Con Crimer (20070 Signify	November 2001	Technical Memorandum			
4. TITLE AND SUBTITLE	November 2001				
4. TITLE AND SUBTILLE		5. FUNDING NUMBERS			
MISSE PEACE Polymers: An In	ternational Space Station				
Environmental Exposure Experi	nent				
-		WU-755-1A-13-00			
6. AUTHOR(S)		W 0-733-1A-13-00			
Kim K. de Groh, Bruce A. Banks	s, Anne Hammerstrom, Erica Y	Youngstrom,			
Carolyn Kaminski, Laura Marx,	Elizabeth Fine, Jonathan D. G	Gummow, and			
Douglas Wright					
7. PERFORMING ORGANIZATION NAME(S	8. PERFORMING ORGANIZATION				
National Agranautics and Space	Administration	REPORT NUMBER			
National Aeronautics and Space					
John H. Glenn Research Center a	E-13113				
Cleveland, Ohio 44135–3191					
9. SPONSORING/MONITORING AGENCY I	10. SPONSORING/MONITORING AGENCY REPORT NUMBER				
National Aeronautics and Space	Administration				
Washington, DC 20546-0001	NASA TM—2001-211311				
<i>z</i> ,	AIAA-2001-4923				
		11111 2001 4723			
11. SUPPLEMENTARY NOTES		I			

Prepared for the International Space Station Utilization-2001 sponsored by the American Institute of Aeronautics and Astronautics, Cape Canaveral, Florida, October 15–18, 2001. Kim K. de Groh and Bruce A. Banks, NASA Glenn Research Center; Anne Hammerstrom, Erica Youngstrom, Carolyn Kaminski, Laura Marx, and Elizabeth Fine, Hathaway Brown School, 19600 North Park Boulevard, Shaker Heights, Ohio 44122; Jonathan D. Gummow, Ohio Aerospace Institute, 22800 Cedar Point Road, Brook Park, Ohio 44142; and Douglas Wright, Cleveland State University, 1983 E. 24th Street, Cleveland, Ohio 44115. Responsible person, Kim K. de Groh, organization code 5480, 216–433–2297.

## Unclassified - Unlimited Subject Categories: 18 and 27 Distribution: Nonstandard Available electronically at <a href="http://gltrs.grc.nasa.gov/GLTRS">http://gltrs.grc.nasa.gov/GLTRS</a> This publication is available from the NASA Center for AeroSpace Information, 301–621–0390.

#### 13. ABSTRACT (Maximum 200 words)

Forty-one different polymers are being exposed to the low Earth orbit (LEO) environment on the exterior of the International Space Station (ISS) for one year as part of MISSE (Materials International Space Station Experiment). MISSE is a materials flight experiment sponsored by the Air Force Research Lab/Materials Lab and the National Aeronautics and Space Administration (NASA). A second set of the same polymers is planned to be flown as part of PEACE (Polymer Erosion And Contamination Experiment), a short duration shuttle flight experiment, and therefore these 41 polymers on ISS are collectively called the MISSE PEACE Polymers. The objective of the MISSE PEACE Polymers experiment is to accurately determine the atomic oxygen (AO) erosion yield of a wide variety of polymeric materials. The polymers range from those commonly used for spacecraft applications, such as Teflon" FEP, to more recently developed polymers, such as high temperature polyimide PMR (polymerization of monomer reactants). Additional polymers were included to explore erosion yield dependence upon chemical composition. Details on the specific polymers being flown, flight sample fabrication, and pre-flight characterization techniques will be discussed. The MISSE PEACE Polymers experiment was placed on the exterior of ISS during a spacewalk on August 16, 2001 and is planned to be retrieved in the fall of 2002. The erosion yield data obtained from this experiment will be compared with data from the future short duration experiment PEACE and with predicted results from models developed by a Canadian group that predicts the AO erosion yield of organic materials based on their chemical structure. Having flight data, and comparing flight data with the predictive model results, will be valuable for spacecraft design purposes.

14.	SUBJECT TERMS	15. NUMBER OF PAGES		
	Atomic oxygen; Internation	17		
	Environmental degradation	16. PRICE CODE		
17.	SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
	Unclassified	Unclassified	Unclassified	